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CANE MILLING OF NICOTINE FROM GREEN NICOTIANA RUSTICA

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CONTENTS

CANE MILLING OF NICOTINE FROM GREEN NICOTIANA RUSTICA

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ABSTRACT

Cane milling of green Nicotiana rustica (var. Brasilia, commonly known as Rustica) in a 10 by 14 inches, three-roller mill expressed 92 percent of the total nicotine originally present with or without addition of lime before pressing. Five pressings were made on green rustica to simulate a plant with five mills in series; water was added (60 pounds per 100 pounds of bagasse) in the last two pressings before milling. Final nicotine content of the juice was 0.4 to 0.5 percent by weight and slightly less than 1 pound of juice was obtained for each pound of rustica fed to the mill. Hourly feed rates were calculated to be 2,500 to 3,000 pounds of green rustica per hour. In addition to nicotine, the juice contained 7.6 percent solids; this was 55 to 60 percent of the original solids in the feed.

INTRODUCTION

After World War II, because of a nicotine shortage, the Eastern Utilization Research and Development Division's laboratory studied several methods[3, 4] for recovering nicotine from Nicotiana rustica (var. Brasilia, commonly known as Rustica). A process was developed which comprised (a) expressing and liming the juice from green Nicotiana rustica plants, (b) extracting the nicotine from the juice with kerosene in a liquid-liquid extraction column, and (c) recovering the nicotine from the kerosene with sulfuric acid as 40-percent nicotine sulfate. A publication[3] primarily covered the second and third steps of this process, but no data on the expressing of the juice were included.

The work though completed years ago has now become of particular interest to investigators in those developing countries faced with the problem of how to protect

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[3]Griffin, E. L., Jr., Phillips, G. W. M., Claffey, J. B., Skalamera, J. J., and Strolle, E. O. Nicotine sulfate from Nicotiana rustica. Ind. Eng. Chem. 44: 274-279. 1952.

[4]Claffey, J. B., Eskew, R. K., and Beinhart, E. G. Recovery of nicotine from Nicotiana rustica by steam distillation. U. S. Dept. Agr. AIC-284, 8 pp. 1950.
_____Strolle, E. O., Phillips, G. W. M., and Eskew, R. K. Scrubbing nicotine from steam. U. S. Dept. Agr. AIC-285, 6 pp. 1950.

farm crops from insect devastation. Utilizing local plants for insecticide sources or using idle equipment normally employed in processing other crops, e.g., cane mill, yields economical and sociological benefits which are obvious. Because of this, the research conducted years ago appears timely. This Division, therefore, has deemed it advisable to make the work on the cane milling of Nicotiana rustica more readily available by assembling the pertinent information in this publication. The information includes a description of the equipment, the experiments carried out, and the experimental data obtained from these experiments.

DESCRIPTION OF EQUIPMENT

The mill employed was a "Squier" motor driven cane mill, 10 by 14 inches, three-roller mill connected to a 2 by 10 inches airdraulic accumulator. The mill rams were 4 inches in diameter, accumulator ratio 25:1, and the ram areas were 25.20 square inches. One pound per square inch air pressure equaled 0.315 tons pressure on the top roll. The load between the three rolls was 1.22 x hydraulic load. 20.50 tons hydraulic load equaled 25 tons loading between rolls. Front roll was on the feed end of the mill; speed of rolls was 22.2 feet per minute. The mill was manufactured by the George L. Squier Manufacturing Co., 490 Broadway, Buffalo, N. Y.

DESCRIPTION OF EXPERIMENTS

Operation of the Mill During Experimentation

Whole rustica plants were cut in a rotary forage mill whose blades were set for a 1/2-inch cut. This cut material was then fed to the cane mill. The front and top rolls were set at 1/8-inch clearance, point to point; the back and top rolls were set metal to metal. The total pressure on the top roll was 28 tons which is the maximum safe operating pressure for this mill. The mill accepted the feed without any rejects and was fed as fast as it would accept feed. The top roll was floating throughout the operations so the maximum pressure was being exerted. Discharge from the mill was a uniformly thick blanket and the expressed juice passed first through a 20-mesh screen and then through a 40-mesh screen. The solid material retained by the screens was mixed into feed of the succeeding pass.

Preliminary Experiments with Subgrade Plants

Two preliminary experiments were made with small lots (80 and 90 pounds) of subgrade plants. These runs showed that such a large quantity of solids was scraped out of the deep grooves that the solids could not be separated from the juice. Hence, the preliminary runs revealed a fundamental difference between milling sugar cane and rustica. With sugar cane the leaves are cut off and only the stalks are fed to the

mill. The stalks are fibrous and do not clog the deep grooves. Sugar extraction is improved by the deep grooves as they afford a free path for the escape of the juice before it is reabsorbed by the cane. However, with rustica, it is necessary to cane mill both the leaves and stalks since about 75 percent of the total nicotine is in the leaves. Because the leaves are pulpy, the grooves become clogged. If the grooves are then cleaned by the deep-groove scrapers, the amount of solids expressed with the juice is too high and the juice loses its fluidity and cannot be handled effectively. In the quantitative experiments discussed in the following section the scrapers were kept inactive.

Quantitative Experiments

Two experiments were made using approximately 500 pounds of rustica per experiment, one with added lime and the other without lime. In both experiments, the plants were cut in a Papec cutter using a 1/2-inch cut, and the mill was operated as described under "Operation of the Mill During Experimentation."

In the experiment without lime, the material was passed through the cane mill three times. Then water was added for the fourth and fifth passes; the amount of water added was 60 pounds of 90° F. water per 100 pounds of bagasse. The amount added (60 pounds) was the maximum amount of water the bagasse would absorb. The five passes, three dry and two wet, correspond to an industrial installation of five three-roll mills in series.

The experiment with added lime was similar to that without lime except that lime was added before the first pressing and the fourth pressing. Amount of lime added for the first pressing was 0.8 pound of lime per 100 pounds of rustica plant; for the fourth, 0.8 pound of lime per 100 pounds of bagasse was added. In the fourth pass, the lime was first mixed with the water to make a slurry which was then added to the bagasse before pressing.

Moisture determinations, in feed and bagasse, were made in a Dietert moisture teller (manufactured by Harry W. Dietert Co., Detroit, Mich.). This is essentially a forced-draft electrically heated oven with special plans having perforated bottoms through which the hot air is blown. A known weight of sample is placed in the pan and dried to constant weight; and from the initial and final weights, moisture is calculated. Nicotine was determined by the Association of Official Agricultural Chemists' method in which the nicotine is precipitated by silico tungstic acid.

RESULTS

For convenience, the results have been arranged in tabular form in tables 1 to 5, inclusive. Table 1 includes all the quantities necessary for an overall material balance for the five pressings taken as a whole. In both runs, as can be seen from items 8 to 11, inclusive, 95 percent of all the materials used can be accounted for. For a pilot-plant experiment of this magnitude this is very good and indicates that the experiments were carefully planned and executed.

TABLE 1.--Overall material balance for all five pressings

Item	No lime added	Lime added
1. Total rustica fed (lb.)	520.50	506.00
2. Total juice obtained (lb.)	508.00	500.00
3. Ratio, juice/feed (lb. juice/lb. initial feed)	0.97	0.99
4. Water added (lb.)	106.00	96.00
5. Final bagasse (lb.)[1]	80.00	75.00
6. Lime added (lb.)	-	4.70
7. Samples (lb.)[2]	6.50	6.0
8. Total input, feed + water + lime	626.5	606.7
9. Total output, juice + bagasse + samples	594.5	581.0
10. Percent total input accounted for	95.0	95.6
11. Percent unaccounted for (losses because of spillage, etc.)	5.0	4.4

[1]Weight is that before sampling.

[2]Weight of samples other than final bagasse.

Table 2 shows the overall nicotine recovery and also the solids expressed along with the juice. As can be seen, over one-half of the solids originally present were in the juice. This was important in the subsequent use of the juice since it had to be filtered before it could be used in the liquid-liquid extraction column to recover the nicotine.

TABLE 2.--Nicotine recovery and solids in juice for all five pressings

Item	No lime added	Lime added
Percent nicotine in feed	0.455	0.523
Pound of nicotine in feed	2.37	2.65
Percent nicotine in juice	0.433	0.49
Pound of nicotine in juice	2.20	2.45
Percent nicotine recovered	92.7	92.3
Percent solids in feed	12.5	13.6
Pound of solids in feed	65.0	69.0
Percent solids in juice	7.7	7.6
Pound of solids in juice	39.0	37.8
Percent original solids in juice	60.0	54.7

Table 3 shows the amounts of feed, liquid, and solid for each pressing; the weight of sample; duration of each pressing; and a calculated hourly feed rate. The data from table 3 are shown in figure 1, a flow sheet of the pressing operation. The amount of feed for each pressing is the bagasse from the previous pressing less the weight of sample taken. Also included in the flow sheet are amounts of water added before the fourth and fifth pressings, the amount of lime added, and total weight of juice obtained.

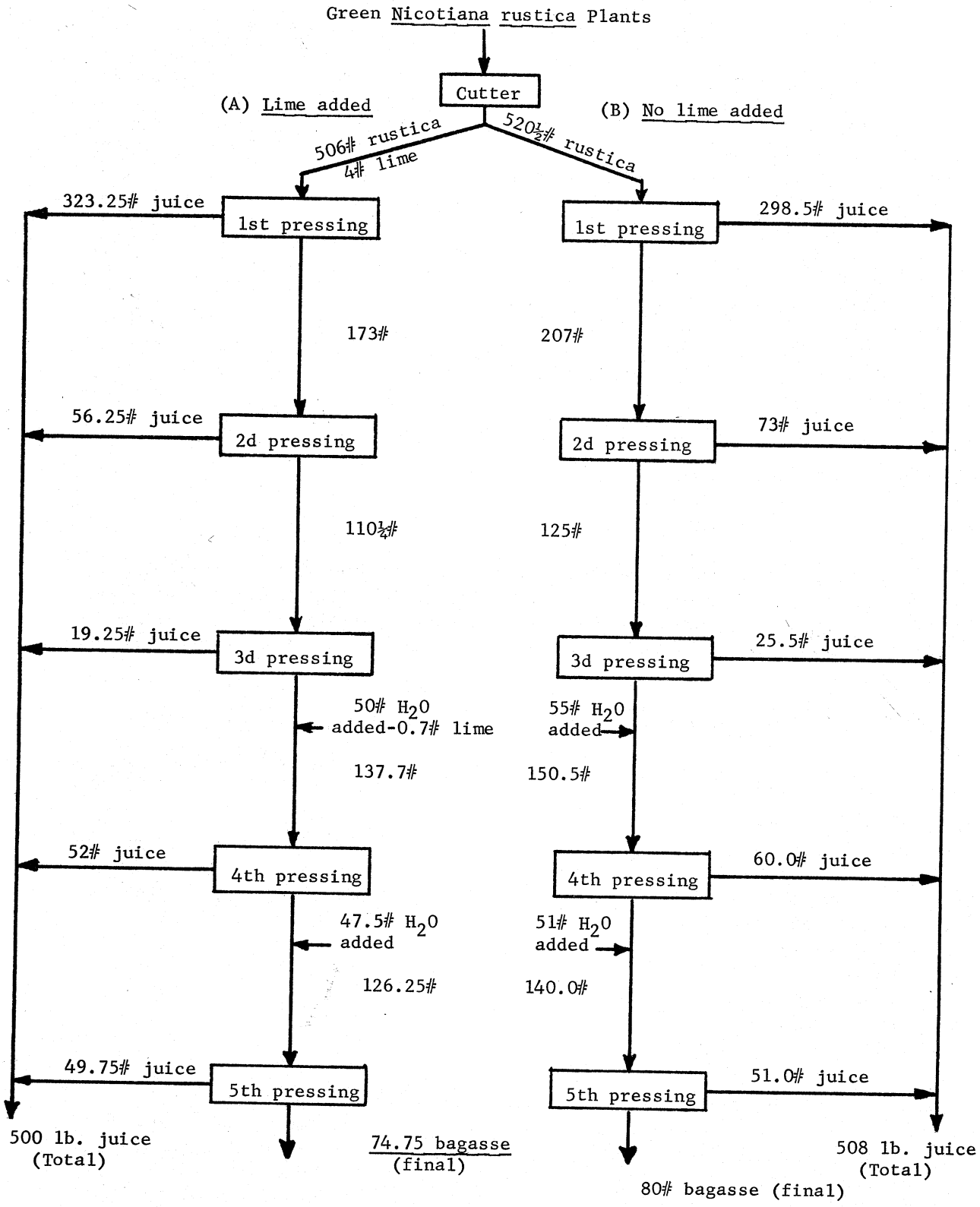

Figure 1. Flow sheet for cane milling green Nicotiana rustica.

TABLE 3.--Feed, juice and bagasse for each stage

Pressing	Feed	Liquid	Solid	Weight sample	Duration of run		Feed rate to mill
	Lb.	Lb.	Lb.	Lb.	Min	Sec.	Lb./hr.
No lime added:							
1st	520.5	298.5	209	2.0	12	5	2,580
2d	207.0	73.0	126.5	1.5	6	48	1,830
3d	125.0	25.5	97.00	1.5	5	31	1,380
4th	150.50[1]	60.0	90.5	1.5	6	30	1,410
5th	140.00[2]	51.0	80.0[3]	6.0	5	58	1,410
Lime added:							
1st	510.00[4]	323.25	174.5	1.5	10	13	3,000
2d	173.00	56.25	111.75	1.5	5	35	1,860
3d	110.25	19.25	88.50	1.5	4	30	1,470
4th	137.70[5]	52.00	80.25	1.5	5	02	1,650
5th	126.25[6]	49.75	74.75[3]	5.25	4	51	1,560

[1] 55 lb. H_2O added.

[2] 51 lb. H_2O added.

[3] Final bagasse.

[4] 4 lb. lime added.

[5] 50 lb. H_2O + 0.7 lb. lime added.

[6] 47.5 lb. H_2O added.

Table 4 shows the percent of the original nicotine recovered at each pressing. The percent nicotine in the juice for each pressing is included in this table and this figure multiplied by the weight of juice (shown in table 3) gives the pounds of nicotine obtained in each pressing. This figure divided by the pounds of nicotine present in the feed times 100 gives the percent recovered. The data in table 4 also reveal that while adding lime does increase nicotine extraction on the first pressing, for all five pressings there is no significant difference between adding lime or not adding lime before pressing.

TABLE 4.--Percent nicotine extracted at each pressing

	No lime added			Lime added		
Pressing	Percent nicotine[1]	Pounds	Percent recovered	Percent nicotine[1]	Pounds	Percent recovered
1st	0.483	1.44	60.8	0.553	1.78	67.0
2d	0.509	0.37	15.6	0.512	0.29	10.9
3d	0.492	0.13	5.5	0.519	0.10	3.8
4th	0.275	0.17	7.2	0.334	0.17	6.4
5th	0.164	0.08	3.4	0.232	0.12	4.5
		Total	92.5		Total	92.6

[1] This is the percent present in the juice for that pressing.

Table 5 shows the energy requirements for operating the mill using the conditions under which these experiments were carried out. The power consumption of the motor driving the cane mill was measured by a Westinghouse Type TA industrial analyzer. The motor driving the mill rolls was a 10 horsepower, 3-phase, 60-cycle, 208 volt, 585 r.p.m. induction motor. Both observed and calculated values of the power consumption are included. Calculations were made using the formula shown in the footnote of table 5.

TABLE 5. Energy requirements for milling

Item	No pressure on rolls	28-ton pressure on top roll No feed	28 tons first pass	28 tons second pass
Amperes per phase	21.5	23.4	24.0	24.2
Volts	210	209	208	208
Power factor	0.34	0.46	0.51	0.52
Kilowatts (observed)	2.50	3.80	4.40	4.60
Kilowatts (calculated)[1]	2.66	3.88	4.40	4.50

[1] Calculated as follows: Volts x amperes x power factor x 1.73 divided by a 1,000.